REPORT DOCUMENTATION PAGE

AFRL-SR-AR-TR-02-

0225

1. AGENCY USE ONLY *(Leave blank)*	2. REPORT DATE	3. REPORT TYPE AND DATES COVERED
	13 Jun 02	FINAL (01 FEB 98 TO 30 JUN 01)

4. TITLE AND SUBTITLE
COMBINED THEORETICAL AND EXPERIMENTAL STUDY OF A NEW MECHANISM OF YIELDING WITH APPLICATION TO THE BRITTLE-DUCTILE TRANSITION.

5. FUNDING NUMBERS
F49620-98-1-0245

6. AUTHOR(S)
PROFESSOR POPE

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
DEPARTMENT OF MATERIALS SCIENCE AND ENGINEERING
UNIVERSITY OF PENNSYLVANIA
PHILADELPHIA, PA 19104

8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
AIR FORCE OFFICE OF SCIENTIFIC RESEARCH
801 N. RANDOLPH STREET
ARLINGTON, VA 22203

10. SPONSORING/MONITORING AGENCY REPORT NUMBER

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION AVAILABILITY STATEMENT

Approved for public release; distribution unlimited.

12b. DISTRIBUTION CODE

13. ABSTRACT *(Maximum 200 words)*
A new strain-rate dependent mechanism of dislocation generation that can become active suddenly above a 'critical temperature has been developed in our research carried out under the present AFOSR support. This mechanism is a thermally driven, stress-assisted cooperative instability of many dislocation loops that leads to an outburst of dislocation activity above the strain-rate dependent critical temperature. The strain-rate dependence originates from the glide of pre-existing and thermally nucleated dislocations below the critical temperature. We have determined theoretically and shown by experiments that the onset of yielding in a crack-free crystal with a very low dislocation content (Si in our study) is remarkably similar to the brittle-to-ductile transition (BDT) in a pre-cracked crystal of the same material. There is significant evidence to show that both processes are controlled by the cooperative process of dislocation generation. As a result, we now have, for the first time, a model that is capable of predicting the brittle-to-ductile-transition-temperature (BDTT) of a material as a function of strain rate and dislocation content.

20020719 137

14. SUBJECT TERMS

15. NUMBER OF PAGES

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
UNCLASSIFIED	UNCLASSIFIED	UNCLASSIFIED	

UNIVERSITY of PENNSYLVANIA

David P. Pope
Professor
Department of Materials Science and Engineering
School of Engineering and Applied Science
Philadelphia, PA 19104-6272
215-898-9837 Fax: 215-573-2128
email: pope@seas.upenn.edu

JUN 13 2002

6/10/02

Craig S. Hartley, Ph.D., P.E.
Program Manager, Metallic Materials
AFOSR/NA
801 North Randolph Street, Room 732
Arlington, VA 22203-1977

RE: Last Annual Report on Grant F49620-98-1-0245, expired June 30, 2001

Dear Craig;

Enclosed is one copy of the last annual report on this grant.

The final report has also been prepared and is now making its way through our University Office of Research Administration on its way to you.

Sincerely;

Dave

David P. Pope

cc: M. Khantha
V. Vitek

COMBINED THEORETICAL AND EXPERIMENTAL STUDY OF A NEW MECHANISM OF YIELDING WITH APPLICATION TO THE BRITTLE-DUCTILE TRANSITION AFOSR GRANT NO.: F49620-98-1-0245

M. Khantha
D. P. Pope
V. Vitek
Department of Materials Science and Engineering
University of Pennsylvania,
Philadelphia, PA 19104.

Abstract

A new strain-rate dependent mechanism of dislocation generation that can become active suddenly above a 'critical' temperature has been developed in our research carried out under the present AFOSR support. This mechanism is a thermally driven, stress-assisted *cooperative instability* of *many* dislocation loops that leads to an outburst of dislocation activity above the strain-rate dependent critical temperature. The strain-rate dependence originates from the glide of pre-existing and thermally nucleated dislocations below the critical temperature. We have determined theoretically and shown by experiments that the onset of yielding in a crack-free crystal with a very low dislocation content (Si in our study) is remarkably similar to the brittle-to-ductile transition (BDT) in a pre-cracked crystal of the same material. There is significant evidence to show that both processes are controlled by the cooperative process of dislocation generation. As a result, we now have, for the first time, a model that is capable of predicting the brittle-to-ductile-transition-temperature (BDTT) of a material as a function of strain rate and dislocation content.

Monte Carlo Simulation of the Cooperative Dislocation Generation - Theory

The purpose of this simulation has been to demonstrate the predictions of the static cooperative dislocation generation model based on the mean field theory (see §2.0) using the Monte Carlo method (Ling 1999, Khantha et al. 1999). However, while the mean field theory has been formulated for the three-dimensional medium with dislocation loops, the Monte Carlo study has been made for the two-dimensional (2D) case and defects involved are dislocation dipoles composed of point dislocations. The relevant mean-field theory of the two-dimensional case is, of course, analogous to the three dimensional case and was developed in the previous phase of our AFOSR-funded research (Khantha et al. 1994, Khantha and Vitek 1997b).

In the Monte Carlo study (Ling 1999) the dislocation dipoles are assumed to form by thermal fluctuations at finite temperatures on a triangular lattice under applied stresses. The full Hamiltonian of the system is employed which means that the interaction between all the

dislocations present in the system is explicitly taken into account. On the other hand, in the mean field approach the energy is evaluated as the energy of a test dislocation loop/dipole present in a medium containing all the other dislocation loops/dipoles and is given by an equation similar to equation (1) with K_0 replaced by K_{eff}. Using the fluctuation-dissipation theorem (Reichl 1980), K_{eff} can be determined from the results of the Monte Carlo study in terms of the auto-correlation function of the plastic strain tensor associated with the dislocation dipoles. The simulation is carried out using a procedure similar to that proposed by Saito (Saito 1982) in the modeling of the Kosterlitz-Thouless transition. The standard Metropolis algorithm is used and there are three basic steps: Creation and annihilation of nearest neighbor dislocation dipoles and expansion/contraction of existing dipoles when dislocations forming a dipole are allowed to move to empty sites. The latter process is essential since it allows dislocation dipoles to expand or shrink under the applied stress. The details of the simulation have been summarized in Khantha et al. (1999).

Figure 1 shows the dependence of the effective energy coefficient K_{eff}, evaluated from the Monte Carlo simulation on temperature for different applied stresses. K_{eff} starts to decrease precipitously at temperatures between 0.2 and 0.25 and this decrease commences at lower temperatures for higher stresses. These results are in very good agreement with the predictions of the mean-field theory and confirm the possibility of a cooperative process of dislocation generation above a critical temperature.

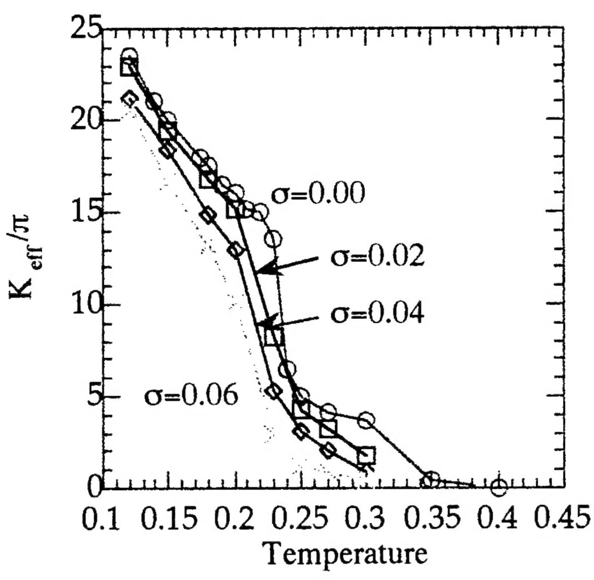


Fig. 1. Effective energy coefficient K_{eff} as a function of temperature for different applied stresses. All quantities are in normalized units.

Experimental Results

The onset of plasticity was measured using three point bend tests. At and above a certain minimum temperature the bending stress reaches a very high value, about 1% of the modulus, and then rapidly drops by a factor of about three as the sample undergoes massive

plastic deformation localized in the center section of the beam under the loading point. If a typical polycrystalline metallic sample containing large numbers of pre-existing dislocations were subjected to this test, the sample would be plastically deformed over much of its entire length, not just in the center. The difference between the two observations is due to the extremely low initial dislocation content in the Si beam. The only plasticity observed in the Si sample is in the region of maximum bending stress near the point of load application.

The minimum temperature at which such massive plasticity is observed, i.e., an upper yield stress followed by continuous flow at a lower yield stress, is experimentally defined here as the BDTT for that strain rate. At low temperatures, the samples show no macroscopic plasticity and brittle fracture ensues at a certain stress level. The transition from fully brittle to ductile behavior is not perfectly sharp, however. In a small temperature range, within a few tens of K below the BDTT, depending on the strain rate, the samples show limited plasticity followed by brittle fracture. In this temperature range large numbers of dislocations are produced, but they are insufficient for sustained flow, and thus the samples fracture in a brittle manner after small amounts of deformation.

The absolute values of the BDTT's that we have measured on Si are in the range of values reported in the literature obtained on pre-cracked samples. Our values of the BDTT range from a low of 885K for the slowest cross head displacement rate of 0.01 mm/min to a high of 1006K at the highest rate of 0.2 mm/min. In comparison, for pre-cracked samples, St. John (St. John 1975) found a BDTT to be between 973 and 1100K, depending on the loading rate, Brede and Haasen (1988) between 937 and 1139K, depending on the loading rate and doping level, and Samuels and Roberts (1989) between 823 and 1073K, again depending on the loading rate. The most important conclusion to be drawn from this comparison is that our measurements of the BDTT made on plain samples fall in the same range as those obtained for pre-cracked samples. Additional data for tests on plain Si samples exist in the literature which are also in agreement with our results. For example, Patel and Chaudhuri (1963) found a BDTT between 900 and 1100K for different loading rates, Pearson et al. (1957) found it to be about 900K for one loading rate and Yonenaga and Sumino (1978) found it to be at least 1073K for all the loading rates they used. Obviously, the values of BDTT obtained on plain and pre-cracked samples are very similar.

We now turn our attention to the temperature region within which there may be substantial plasticity but the sample nonetheless fails in a brittle manner. This region was seen for every loading rate used in our study, and we believe that the discovery of this regime in the plastic behavior of Si is one of the most important results of the study. The fact this region exists shows that there are important differences in the nature of dislocation activity just below and above the BDTT. A quantitative measure of this difference can be seen by plotting the logarithm of the upper yield stress as a function of the inverse test temperature below and above the BDTT, as shown in Figure 2. In both regimes, the variation of the upper yield stress, $\tau_{U.Y.}$, can be expressed in the form

$$\tau_{U.Y.} = c(\dot{\varepsilon})^{1/n} \exp(U/k_B T) \qquad (1)$$

where $\tau_{U.Y.}$ refers to the upper yield stress, $\dot{\varepsilon}$, the strain-rate, n, the strain-rate exponent, U, an apparent activation energy, k_B, the Boltzmann constant, T, the temperature and c is a constant. The observations at all strain-rates show that the value of U is different below and above the BDTT. This is one of the most significant results obtained in our study. The value of U is about 25% lower below the BDTT than above the BDTT. Similar experiments carried out by others usually contain information only above the BDTT (Alexander and Haasen 1968, Alexander 1986). In this regime, our results are in good agreement with both observations and the theoretical model of Alexander and Haasen which is based on the dislocation velocity being controlled by a thermally activated process and a power-law function of the stress. The model also assumes a simple dislocation multiplication rule, as would be the case for Frank-Read sources, and predicts that above the BDTT, the product nU is nearly equal to the activation energy associated with dislocation motion, namely, U_m. We have determined n = 2.7 from our data and using U = 0.77 eV, the value above the BDTT (see Fig. 2), we obtain U_m = 2.06 eV, in excellent agreement with the value reported for intrinsic Si (Alexander 1986). Our data for the variation of the flow stress with inverse temperature is also in good agreement with the predictions of the Alexander and Haasen model.

The existence of the intermediate region is strong evidence in support of our idea that the pre-existing dislocations facilitate the thermal nucleation of dislocation loops at temperatures below the BDTT, and these thermally nucleated dislocations then contribute to plasticity below the BDTT. As will be shown later in this section, the region of the beam that undergoes the plastic bending shows dramatic changes in dislocation density just below and at the point of maximum load.

Post-deformation dislocation densities have been observed using etch-pit techniques and an SEM. These observations were made for a given strain-rate at several temperatures below and above the BDTT and at various stress levels by interrupting the loading at different points along the load-displacement curve, see Figure 3. This figure shows a side surface of two beams after testing. The top half of the beam is in compression and the bottom half is in tension when under load. The contact stresses between the loading point and the top surface of the beam are such that the resolved shear stress promoting <110>{111} dislocation activity in response to the bending stresses is higher on the bottom side of the beam, and therefore massive dislocation nucleation is always seen first on the bottom side of the beam. At 60% of the peak load slip bands are seen to emanate from the load point, see the vee-shaped pattern of dislocation etch pits emanating from the top center of Figure 3a, and in some cases, there is a visible dent under the load point. These slip bands result apparently from contact stresses between the sample and the loading point and appear to be unrelated to the bending stresses. This plastic denting causes non-linear displacements of the load point and is, we believe, the source of most of the nonlinear behavior seen at loads below the peak. A few slip bands (much lower in density compared to Fig. 3b) appear in the bottom half of the beam beginning at about 80% of the maximum load in samples that exhibit an upper yield followed by a load drop

but then fail in a brittle manner. However, at the BDTT there is massive dislocation nucleation in the bottom half of the beam in the dislocation-free region between the slip bands due to contact stresses, see Figure 3b. The generation of these massive slip bands demonstrates a highly nonlinear dislocation nucleation event in regions well-separated from

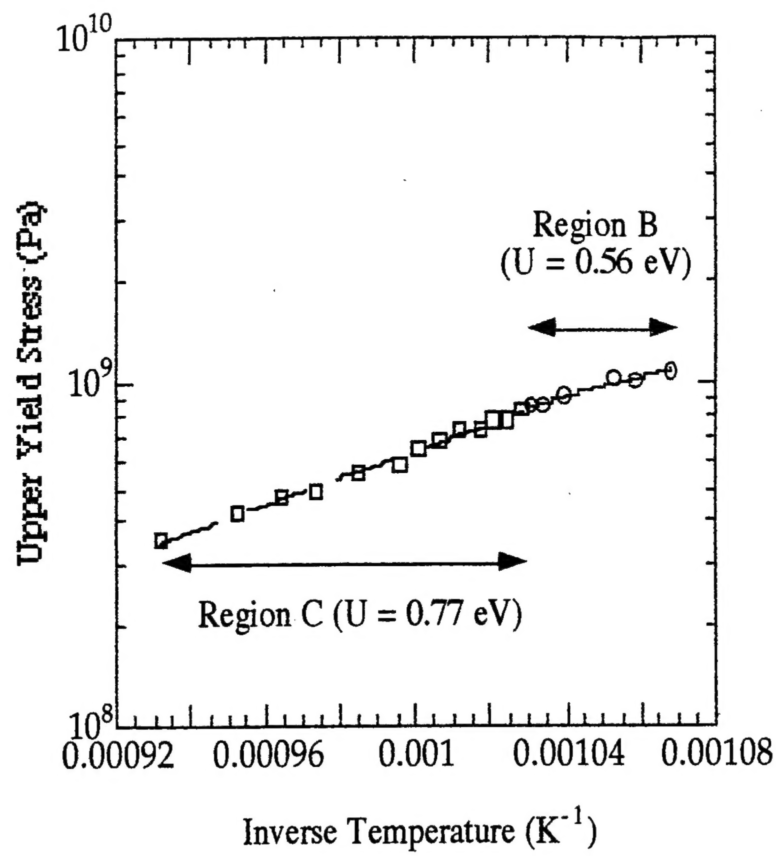
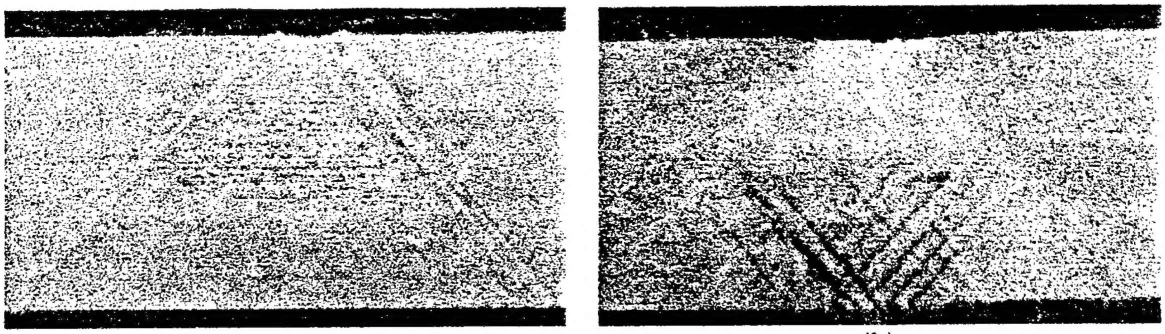


Fig. 2. A plot of the peak stress vs. reciprocal temperature for samples deformed at a rate of 0.1mm/min. Note that the slope of the line below the BDTT is about 25% less than above it.

the slip bands seen in Figure 3a. This dislocation activity then very quickly spreads throughout the thickness of the entire sample. The massive dislocation nucleation therefore appears to be totally unrelated to the slip bands emanating from the point of loading and we conclude that they result from the type of instability described by the theory.

Analysis of the Experimental Results

The experimental observations described in this section are in qualitative agreement with the model. According to this model, the BDTT is determined by the plastic strain contributions arising from the motion of three populations of dislocations: pre-existing dislocations, thermally nucleated dislocations that are nucleated in a x plastic strain contributions arising from the motion of three populations of dislocations: pre-



(a) (b)

Fig. 3. Dislocations revealed by etch pitting on (a) a sample loaded to about 60% of the peak load just below the BDTT and (b) a sample loaded slightly beyond the peak load just above the BDTT. Note the slip bands emanating from the loading point in (a) and massive slip band formation in the central region on the lower side of the beam in (b).

existing dislocations, thermally nucleated dislocations that are nucleated in a temperature interval 50-100K below BDTT, and atomic-size dislocation loops which appear in large numbers only above the BDTT. Massive plasticity can occur only when the atomic-size dislocation loops can expand without any energy barrier, and the temperature at which this instability is triggered is identified with the BDTT. However, considerable dislocation activity can arise in a temperature interval 50-100K below the BDTT due to thermal nucleation of dislocation loops, even in the absence of pre-existing dislocations. This process is a highly non-linear function of the stress, due to the cooperative interactions between dislocations. We believe that the thermally nucleated dislocations play the dominant role in region B. According to the model, the energy barrier for thermal nucleation decreases dramatically with temperature when approaching the BDTT. Thus, we expect that samples will show an upper yield followed by appreciable load-drops just below the BDTT in accordance with the observations. The prediction of the dependence of the BDTT on the loading rate is in excellent agreement with observations as shown in Table 1 below.

Table 1. Comparison of measured and theoretical BDTT values for silicon.

Loading rate (mm/min)	BDTT (K) (Exp.)	BDTT (K) (Theory)
0.01	885	883
0.05	938	938
0.1	972	962
0.2	1006	989

Acknowledgement/Disclaimer

This work was sponsored (in part) by the Air Force Office of Scientific Research, USAF, under grant/contract number F49620-98-1-0245. The views and conclusions contained herein are those of the authors and should not be interpreted as necessarily representing the official policies or endorsements, either expressed or implied, or the Air Force Office of Scientific Research or the US Government.

References

Alexander, H., 1986, *Dislocations in Solids*, edited by Nabarro, F. R. N., (Amsterdam: North Holland) Vol. **7**, p. 115.

Alexander, H. and Haasen, P., 1968, *Solid State Physics*, edited by Seitz, F., Turnbull, D. and Ehrenreich, H., (New York: Academic Press) Vol. **22**, p. 28.

Brede, M. and Haasen, P., 1988, *Acta Metall.* **36,** 2003.

Khantha, M., Ling, M. and Vitek, V., 1999, *Mat. Sci. Res. Int.* **5,** 234.

Khantha, M., Pope, D. P. and Vitek, V., 1994, *Phys. Rev. Lett.* **73,** 684.

Khantha, M., Pope, D. P. and Vitek, V., 1997, *Acta Mater.* **45,** 4687.

Ling, M., 1999, PhD thesis, Univ. of Pennsylvania.

Patel, J. R. and Chaudhuri, A. R., 1963, *J. Appl. Phys.* **34,** 2788.

Pearson, G. L., Read Jr., W. T. and Feldmann, W. L., 1957, *Acta Metall.* **5,** 181.

Reichl, L. E., 1980, *A Modern Course in Statistical Physics,* (Austin: Univ. of Texas Press).

Saito, Y., 1982, *Phys. Rev. B* **26,** 6239.

Samuels, J. and Roberts, S. G., 1989, *Proc. R. Soc. Lond.* **A 421,** 1.

St. John, C., 1975, *Philos. Mag.* **32,** 1193.

Yonenaga, I. and Sumino, K., 1978, *Phys. Stat. Sol. (a)* **50,** 685.

List of Publications and Patents

1. M. Khantha, V. Vitek and D. P. Pope: Strain-rate dependence of the brittle-to-ductile transition temperature in TiAl, in *High-Temperature Ordered Intermetallic Alloys - IX*, edited by J. H. Schneibel, K. Hemker, R. Noebe, S. Hanada and G. Sauthoff, MRS Symposium Proceedings,Vol. 646, p. N1.11, 2001.

2. M. Khantha, V. Vitek and D. P. Pope, "Strain-rate dependent mechanism of cooperative dislocation generation: Application to the brittle-ductile transition". Mat. Sci. Eng. A319-321 (2001) 484-489.

3. Robert H. Folk II, "The Brittle to Ductile Transition in Silicon: Evidence of a Critical Yield Event", PhD Thesis, University of Pennsylvania (2000).

No patents were obtained as part of this program.

Personnel Supported on Grant

1. Robert H. Folk, II, student, received Ph.D. in 2000.
2. M. Khantha, Faculty, Univ. of PA
3. V. Vitek, Faculty, Univ. of PA
4. D. P. Pope, Faculty, Univ. of PA